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Indian Standard

DETERMINATION OF ALUMINIUM AND BARIUM IN WATER BY DIRECT NITROUS OXIDE-ACETYLENE FLAME ATOMIC ABSORPTION SPECTROMETRY

ICS 13.060.50

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

April 2003 **Price Group 2**

Drinks and Carbonated Beverages Sectional Committee, FAD 14

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Drinks and Carbonated Beverages Sectional Committee had been approved by the Food and Agriculture Divisional Council.

This standard is based on direct nitrous oxide-acetylene flame atomic absorption spectrometry to detect micro quantities of aluminium and barium in waters. This method is used for the determination of these elements in packaged waters (packaged natural mineral water as per IS 13428 and packaged drinking water as per IS 14543). In the preparation of this standard, considerable assistance has been derived from standard methods for the examination of water and wastewater published by American Public Health Association, Washington, USA, 20th edition, 1998.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

DETERMINATION OF ALUMINIUM AND BARIUM IN WATER BY DIRECT NITROUS OXIDE-ACETYLENE FLAME ATOMIC ABSORPTION SPECTROMETRY

1 SCOPE

This standard prescribes the determination of aluminium and barium in bottled water by Direct Nitrous Oxide-Acetylene Flame Method

2 APPARATUS

2.1 Atomic Absorption Spectrometer and Associated Equipment

2.1.1 *Atomic Absorption Spectrometer*

Consisting of a light source emitting the line spectrum of an element (hollow-cathode lamp or electrodeless discharge lamp), a device for vaporizing the sample (usually a flame), a means of isolating an absorption line (monochromator or filter and adjustable slit), and a photoelectric detector with its associated electronic amplifying and measuring equipment.

2.1.2 *Burner*

The most common type of burner is a premix, which introduces the spray into a condensing chamber for removal of large droplets. The burner is to be fitted with a special head for use with nitrous oxide and acetylene.

2.1.3 *Readout*

Most instruments are equipped with either a digital or null meter readout mechanism. Most modern instruments are equipped with microprocessors or stand-alone control computer capable of integrating absorption signals over time and linearizing the calibration curve at high concentrations.

2.1.4 *Lamps*

Use either a hollow-cathode lamp or an electrodeless discharge lamp (EDL). Use one lamp for each element being measured. Multi-element hollow-cathode lamps generally provide lower sensitivity than single-element lamps. EDLs take a longer time to warm up and stabilize.

2.1.5 *Pressure-Reducing Valves*

Maintain supplies of fuel and oxidant at pressures somewhat higher than the controlled operating pressure of the instrument by using suitable reducing valves. Use a separate reducing valve for each gas.

2.1.6 *Vent*

Place a vent about 15 to 30 cm above the burner to remove fumes and vapors from the flame. This precaution protects laboratory personnel from toxic vapors, protects the instrument from corrosive vapors, and prevents flame stability from being affected by room drafts. A damper or variable-speed blower is desirable for modulating air flow and preventing flame disturbance. Select blower size to provide the air flow recommended by the instrument manufacturer. In laboratory locations with heavy particulate air pollution, use clean laboratory facilities such as commercially available laminar-flow, clean-air benches or custom-designed work stations and analyze blanks that reflect the complete procedure.

2.2 Nitrous Oxide Burner Head

Use special burner head as suggested in manufacturer's manual. At roughly 20 min intervals of operation it may be necessary to dislodge the carbon crust that forms along the slit surface with a carbon rod or appropriate alternative.

2.3 T-junction Valve or Other Switching Valve

For rapidly changing from nitrous oxide to air, so that flame can be turned 'ON' or 'OFF' with air as oxidant to prevent flashbacks.

3 REAGENTS

3.1 Air

Cleaned and dried through a suitable filter to remove oil, water and other foreign substances. The source may be a compressor or commercially bottled gas.

3.2 Acetylene, Standard Commercial Grade

Acetone, which always is present in acetylene cylinders, can be prevented from entering and damaging the burner head by replacing a cylinder when its pressure has fallen to 689 kPa (100psi) acetylene.

3.3 Metal-Free Water

Use metal-free water for preparing all reagents and calibration standards and as dilution water. Prepare metal-free water by deionizing tap water and/or by using one of the following processes, depending on the metal concentration in the sample: single distillation, redistillation or sub-boiling. Always check deionized or distilled water to determine whether the element of interest is present in trace amounts.

Note — The source water contains Hg or other volatile metals, single or redistilled water may not be suitable for trace analysis because these metals distill over with the distilled water. In such cases, use sub-boiling to prepare metal-free water.

3.4 Hydrochloric Acid, HCl — 1N, 50 percent and conc.

3.5 Nitric Acid, HNO_3 — conc.

3.6 Sulfuric Acid, H_2SO_4 — One percent v/v.

3.7 Hydrofluoric Acid, HF, 1N

3.8 Nitrous Oxide (Commercially Available Cylinders)

Fit nitrous oxide cylinder with a special nonfreezable regulator or wrap a heating coil around an ordinary regulator to prevent flashback at the burner caused by reduction in nitrous oxide flow through a frozen regulator. (Most modern atomic absorption instruments have automatic gas control systems that will shut down a nitrous oxide-acetylene flame safely in the event of a reduction in nitrous oxide flow rate).

Caution — Use nitrous oxide with strict adherence to manufacturer's directions. Improper sequencing of gas flows at startup and shutdown of instrument can produce explosions from flashback.

3.9 Potassium Chloride Solution

Dissolve 250 g KCl in water and dilute to 1 000 ml.

3.10 Aluminium Nitrate Solution

Dissolve 139 g $Al(NO_3)_3.9H_2O$ in 150 ml water. Acidify slightly with cone HNO_3 to preclude possible hydrolysis and precipitation. Warm to dissolve completely. Cool and dilute to 200 ml.

3.11 Standard Metal Solutions

Prepare a series of standard metal solutions in the optimum concentration ranges by appropriate dilution of the stock metal solutions with water containing 1.5 ml conc. HNO_3/l. Stock standard solutions are available from a number of commercial suppliers. Alternatively, prepare as described in **3.11.1** and **3.11.2**.

3.11.1 *Aluminium*

Dissolve 0.100 g aluminium metal in an acid mixture of 4 ml 1 + 1 HCl and 1 ml conc. HNO_3 in a beaker. Warm gently to effect solution. Transfer to a 1 litre flask, add 10 ml 1+1 HCl, and dilute to 1 000 ml with water; 1.00 ml = 100 µg aluminium.

3.11.2 *Barium*

Dissolve 0.151 6 g $BaCl_2$ (dried at 250° for 2 h), in about 10 ml water with 1 ml 1 + 1 HCl, add 10.0 ml 1+ 1 HCl and dilute to 1 000 ml with water; 1.00 ml = 100 µg barium.

4 PROCEDURE

4.1 Sample Preparation

Required sample preparation depends on metal form being measured. When determining aluminium or barium, mix 2 ml KCl solution into 100 ml sample or standard before aspiration. Drinking water, may be analyzed directly by atomic absorption spectroscopy for total metals without digestion. On collection acidify such samples to pH<2 with concentrated nitric acid (1.5 ml HNO_3 is usually adequate for drinking water) and analyze directly. For further verification or if changes in existing matrices are encountered, compare digested and undigested samples to ensure comparable results. The digestion procedure is outlined in Annex A. For all samples, make certain that the concentrations of acid and matrix modifiers are the same in both samples and standards.

4.2 Instrument Operation

Because of differences between makes and models of atomic absorption spectrometers, it is not possible to formulate instructions applicable to every instrument. See manufacturer's operating manual. In general, proceed as prescribed in **4.2.1** to **4.2.4**.

4.2.1 Install a hollow cathode lamp for the desired metal in the instrument and roughly set the wavelength dial according to Table 1. Set slit width according to manufacturer's suggested setting for the element being measured. Turn on instrument, apply to the hollow cathode lamp the current suggested by the manufacturer, and let instrument warm up until energy source stabilizes, generally about 10 to 20 min. Readjust current as necessary after warm-up. Optimize wavelength by adjusting wavelength dial until optimum energy gain is obtained. Align lamp in accordance with manufacturer's instructions.

Table 1 Atomic Absorption Concentration Range with Direct Aspiration Atomic Absorption
(*Clause* 4.2.1)

Element	Wavelength, nm	Flame Gases	Instrument Detection Limit, mg/l	Sensitivity, mg/l	Optimum Concentration Range, mg/l
(1)	(2)	(3)	(4)	(5)	(6)
Al	309.3	N-Ac	0.1	1	5-100
Ba	553.6	N-Ac	0.03	0.4	1-20

4.2.2 After adjusting wavelength, install a nitrous oxide burner head. Turn on acetylene (without igniting flame) and adjust flow rate to value specified by manufacturer for a nitrous oxide-acetylene flame, turn 'OFF' acetylene. With both air and nitrous oxide supplies turned 'ON', set T-junction valve to nitrous oxide and adjust flow rate according to manufacturer's specifications. Turn switching valve to the air position and verify that flow rate is the same. Turn acetylene on and ignite to a bright yellow flame. With a rapid motion, turn switching valve to nitrous oxide. The flame should have a red cone above the burner. If it does not, adjust fuel flow to obtain red cone. After nitrous oxide flame has been ignited, let burner come to thermal equilibrium before beginning analysis.

4.2.3 Aspirate a blank consisting of deionized water containing 1.5 ml concentrated HNO_3/l and check aspiration rate. Adjust if necessary to a rate between 3 and 5 ml/min. Zero the instrument. Aspirate a standard of the desired metal with a concentration near the midpoint of the optimum concentration range and adjust burner (both horizontally and vertically) in the light path to obtain maximum response. Aspirate blank again and re-zero the instrument. The instrument now is ready to run standards and samples.

4.2.4 To extinguish flame, turn switching valve from nitrous oxide to air and turn off acetylene. This procedure eliminates the danger of flashback that may occur on direct ignition or shutdown of nitrous oxide and acetylene.

4.3 Standardization

Select at least three concentrations of standard metal solutions (prepared as in **3.11**) to bracket the expected metal concentration of a sample. Aspirate each in turn into the flame and record absorbances. Most modern instruments are equipped with microprocessors and digital readout which permit calibration in direct concentration terms. If instrument is not so equipped, prepare a calibration curve by plotting on linear graph paper absorbance of standards versus concentration. Plot calibration curves for aluminium and barium, based on original concentration of standard before adding KCl solution.

4.4 Analysis of Samples

Rinse atomizer by aspirating water containing 1.5 ml conc. HNO_3/l and zero instrument. Atomize a sample and determine its absorbance.

5 CALCULATIONS

Calculate concentration of each metal ion in µg/l by referring to the appropriate calibration curve prepared according to **4.3**.

Alternatively, read the concentration directly from the instrument readout if the instrument is so equipped. If sample has been diluted, multiply by the appropriate dilution factor.

ANNEX A

(*Clause* 4.1)

NITRIC ACID DIGESTION

A-1 APPARATUS

A-1.1 Hot Plate

A-1.2 Conical (Erlenmeyer) Flasks (125 ml) or **Griffin Beakers** (150 ml, acid washed and rinsed with water)

A-1.3 Volumetric Flasks — 100 ml.

A-1.4 Watch Glasses — Ribbed and unribbed.

A-2 REAGENT

A-2.1 Nitric Acid, HNO_3 —Concentrated analytical or trace metal grade.

A-3 PROCEDURE

Transfer a measured volume (100 ml recommended) of well-mixed, acid preserved sample appropriate for the expected metal concentrations to a flask or beaker. In a hood, add 5 ml concentrated HNO_3. If a beaker is used, cover with a ribbed watch glass to minimize contamination. Boiling chips, glass beads, or Hengar granules may be added to aid boiling and minimize spatter when high concentration levels are being determined. Bring to a slow boil and evaporate on a hot plate to the lowest volume possible (about 10 ml to 20 ml) before precipitation occurs. Continue heating and adding concentrated HNO_3 as necessary until digestion is complete as shown by a light coloured, clear solution. Do not let sample dry during digestion.

Wash down flask or beaker walls and watch glass cover (if used) with metal-free water and then filter if necessary. Transfer filtrate to a 100 ml volumetric flask with two 5 ml portions of water, adding these rinsings to the volumetric flask. Cool, dilute to mark, and mix thoroughly. Take portions of this solution for required metal determinations.

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